

## WG4 activity - NECTAR survey 2

### KEY PARAMETERS FOR POTENTIOMETRIC AND SPECTROPHOTOMETRIC EXPERIMENTS

Instruments, experimental conditions and data treatment procedures are key factors to define reliable equilibrium constant values. It is necessary to know many of these parameters in order to replicate the experiment.

Assuming you need/want to share raw data with the scientific community,  
**which parameters do you consider necessary to completely define the experiment?**

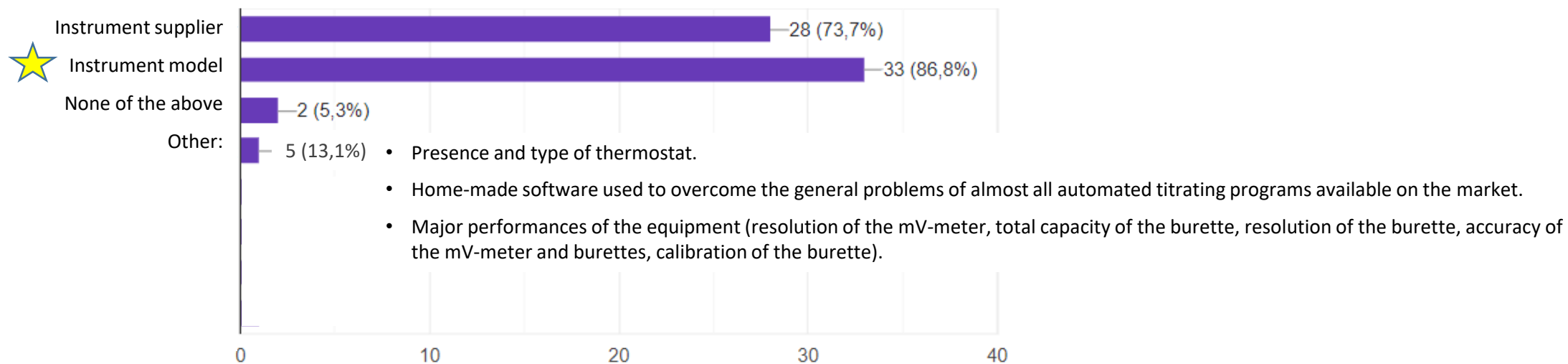


*Parameters with more than 80% responses*

## WG4 activity - NECTAR survey 2 - Answers

### Potentiometry - Instrument - Automated titrator

What information is necessary to describe the measurement system used?



## WG4 activity - NECTAR survey 2 - Answers

### Potentiometry - Instrument - Dosing device

What information is necessary to describe the dosing device used?

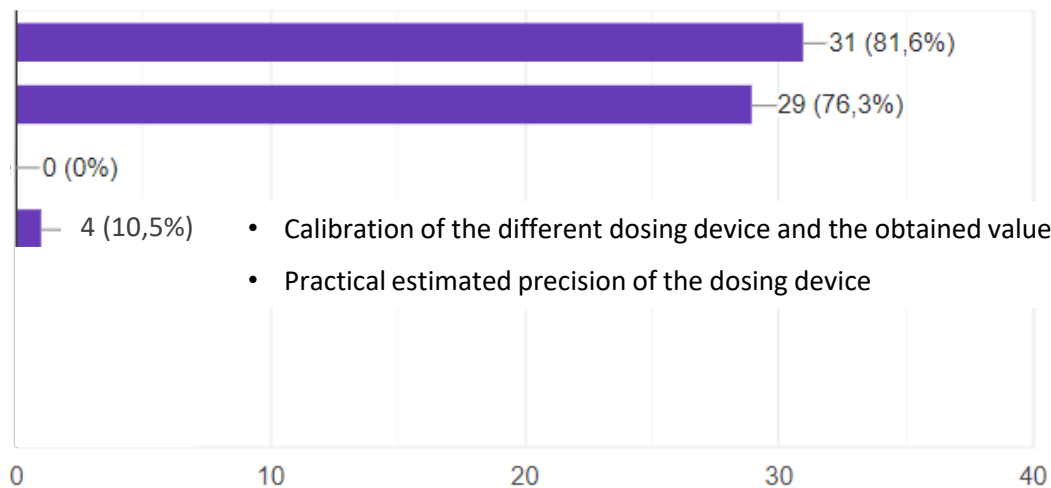
★ Type of dosing device (automated burette, pipette, manual micro-syringe...)

Precision of dosing device (dispenser total volume/steps number)

None of the above

Other: 4 (10,5%)

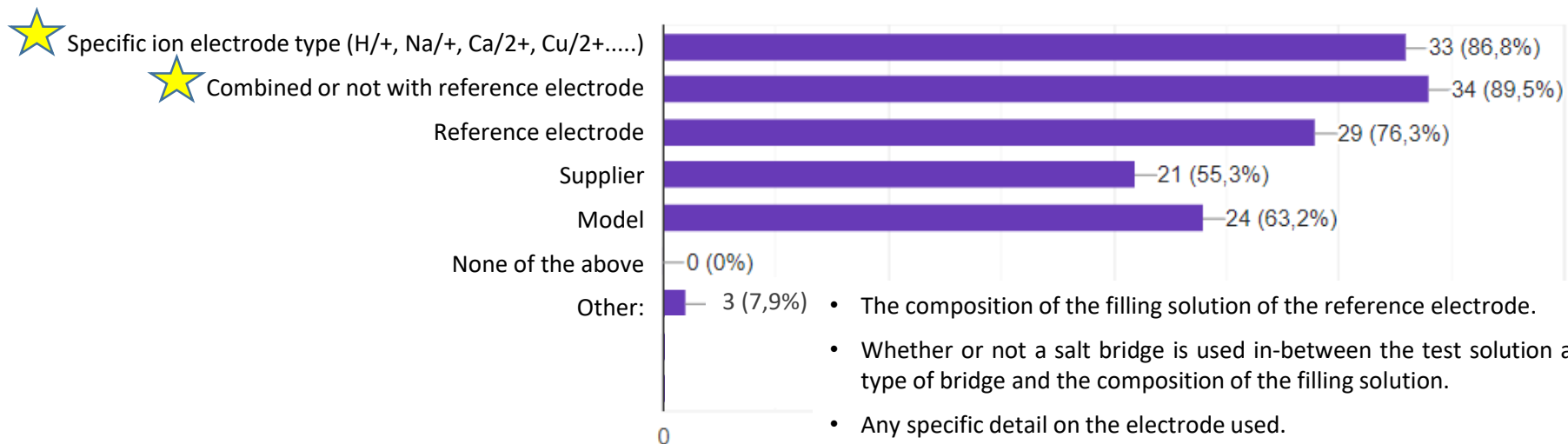
- Calibration of the different dosing device and the obtained values
- Practical estimated precision of the dosing device



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### Potentiometry - Instrument - Electrode

What information is necessary to describe the electrode used?

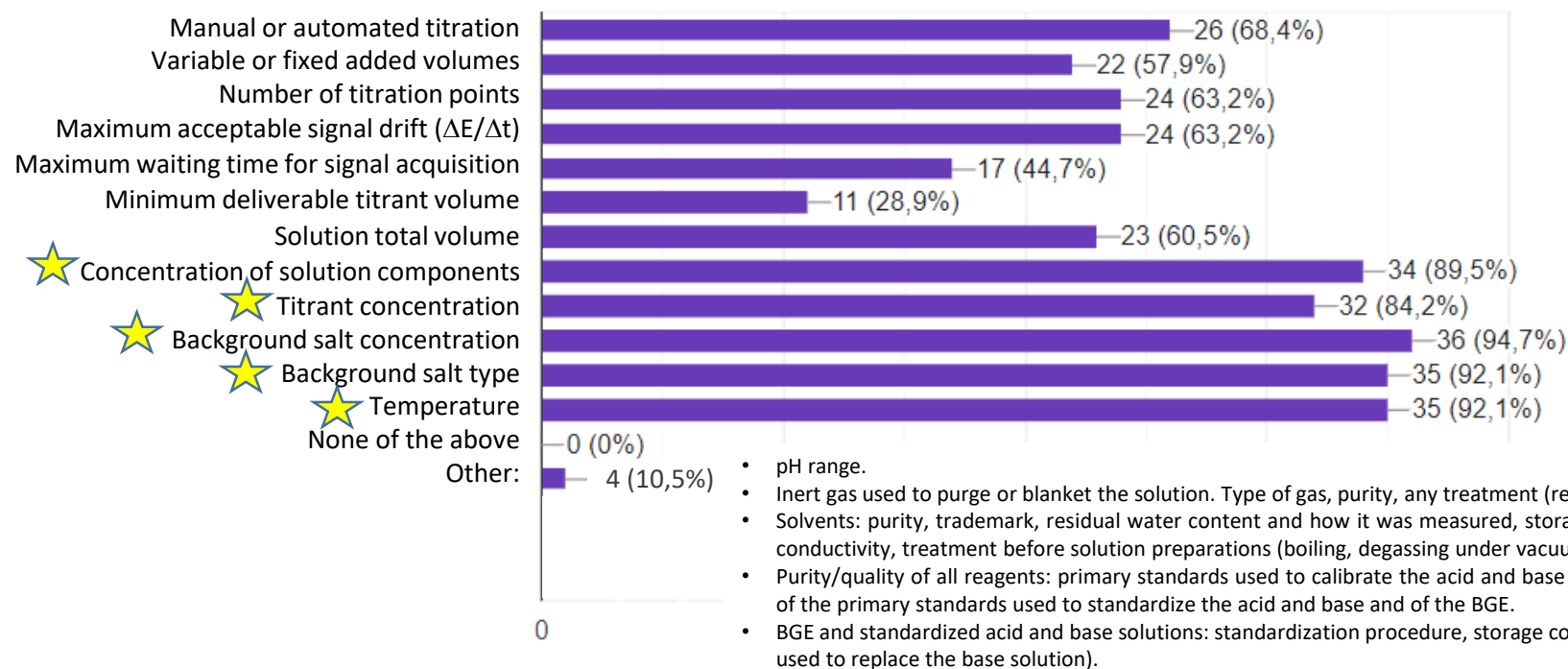


- The composition of the filling solution of the reference electrode.
- Whether or not a salt bridge is used in-between the test solution and the reference electrode. If so, specify the type of bridge and the composition of the filling solution.
- Any specific detail on the electrode used.
- For titrations in non-aqueous solvents or solvent-rich mixtures with water, the electrode preconditioning procedure (example, soaked for one hour in the solvent prior to the calibration) and storage conditions in between measurements (these parameters can influence the stability of the electrode).

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### Potentiometry - Calibration - Electrode calibration in concentration unit

What information is necessary to describe the calibration procedure used?



## WG4 activity - NECTAR survey 2 - Answers

### Potentiometry - Calibration - Electrode calibration in concentration unit

What information is necessary to describe the calibration data treatment?

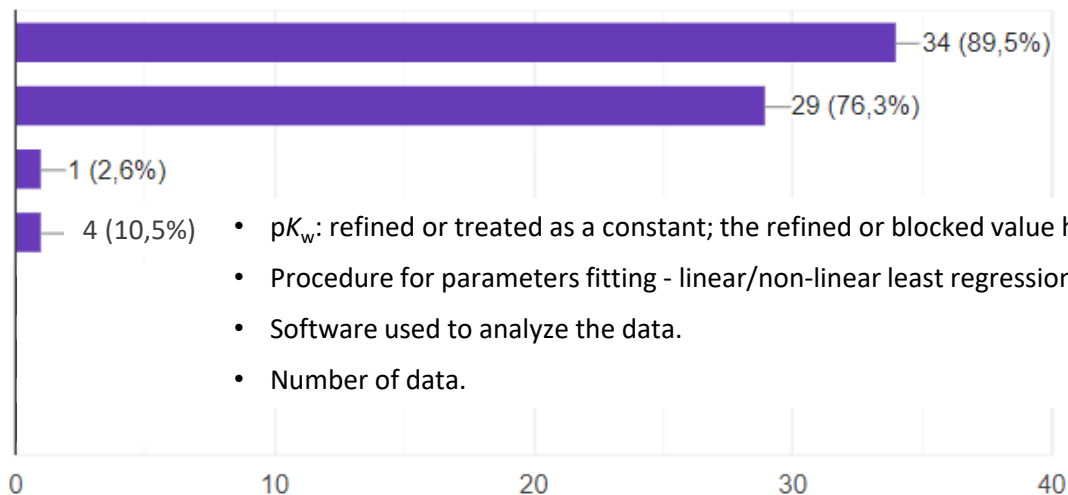


List of optimized Nernst's equation parameters ( $E^0$ , slope, junction potentials,  $pK_w$ )

pH range of the fitted titration points

None of the above

Other:

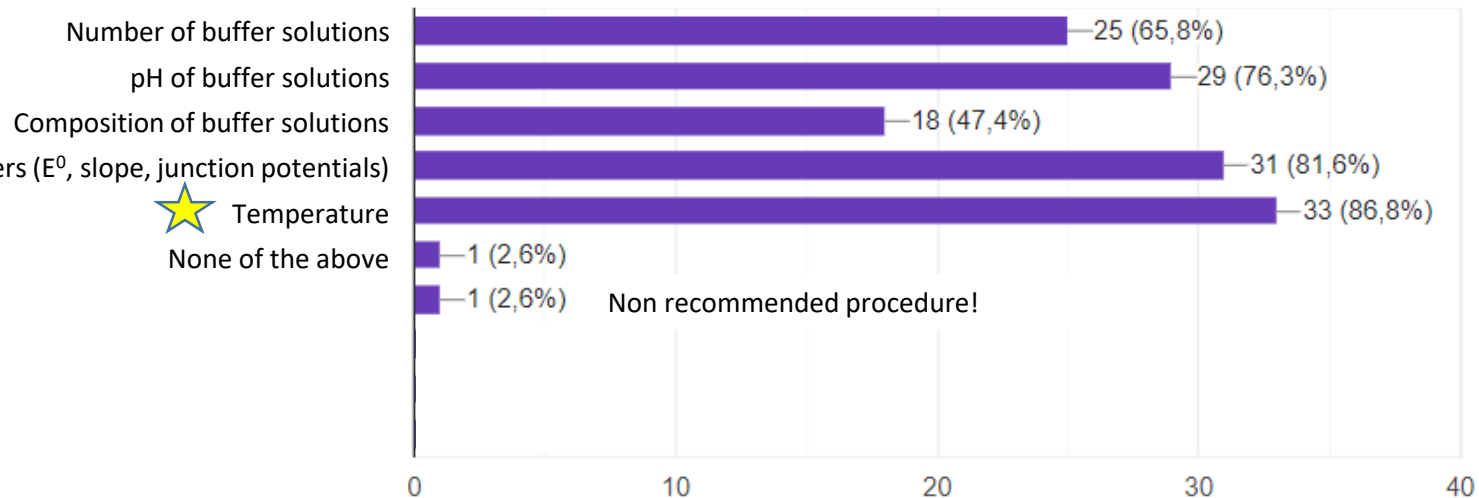


- $pK_w$ : refined or treated as a constant; the refined or blocked value has to be reported.
- Procedure for parameters fitting - linear/non-linear least regression.
- Software used to analyze the data.
- Number of data.

## WG4 activity - NECTAR survey 2 - Answers

### Potentiometry - Calibration - Electrode calibration in activity unit

What information is necessary to describe the calibration procedure used?



List of optimized Nernst's equation parameters ( $E^0$ , slope, junction potentials)

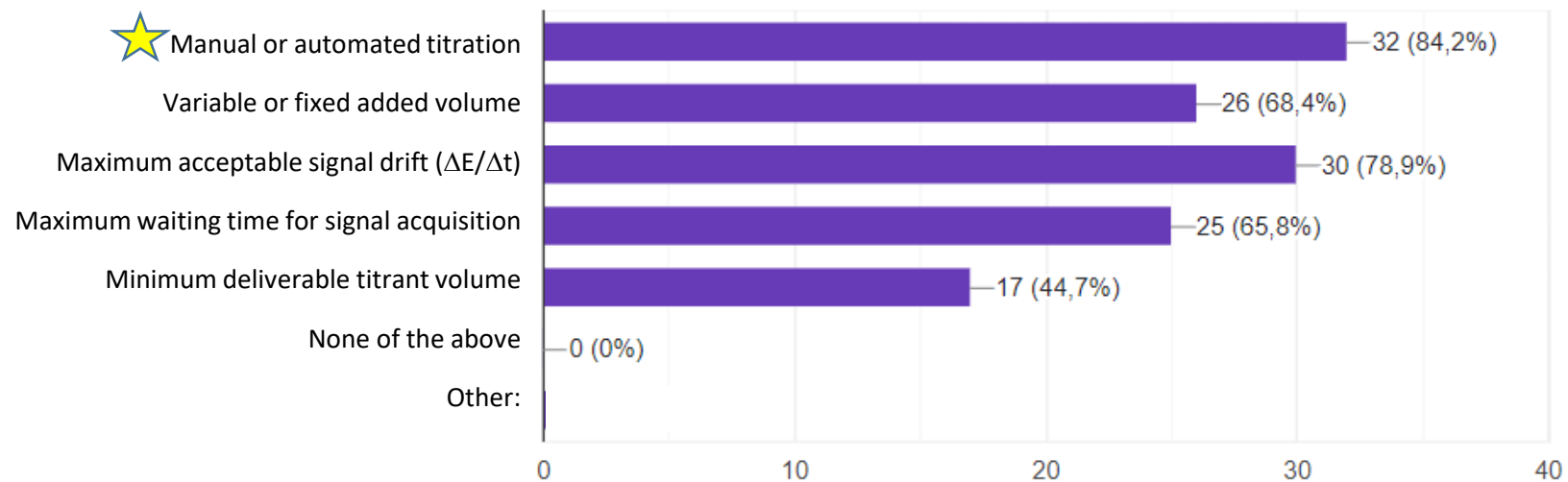


Temperature

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### Potentiometry - Titration procedure

What instrumental information is necessary to describe the experiment?

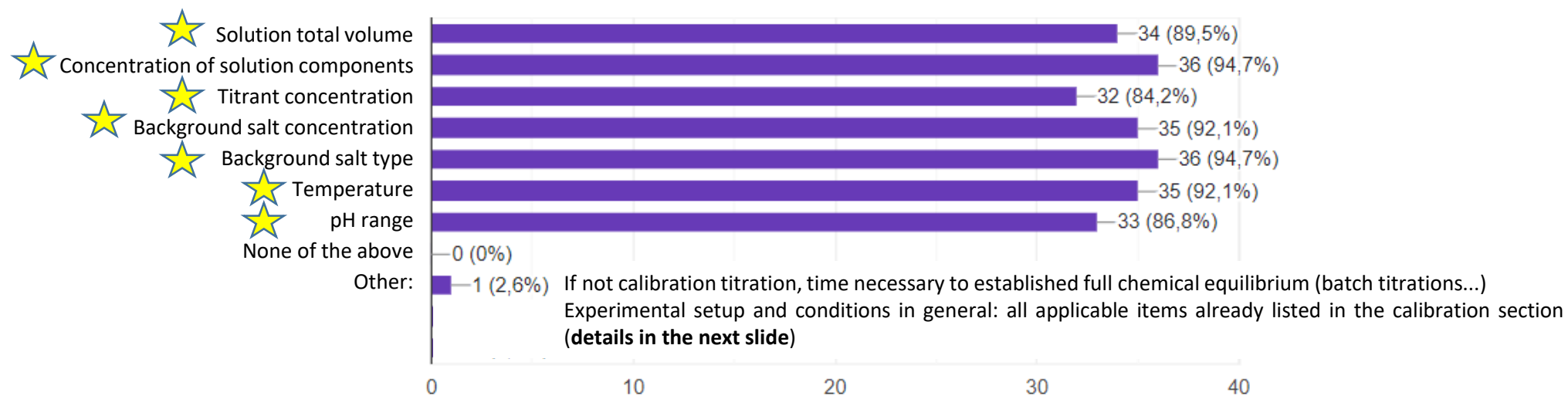




## WG4 activity - NECTAR survey 2 - Answers

### Potentiometry - Titration procedure

What information is necessary to describe the titration procedure used?



## WG4 activity - NECTAR survey 2 - Answers

### Potentiometry - Titration procedure

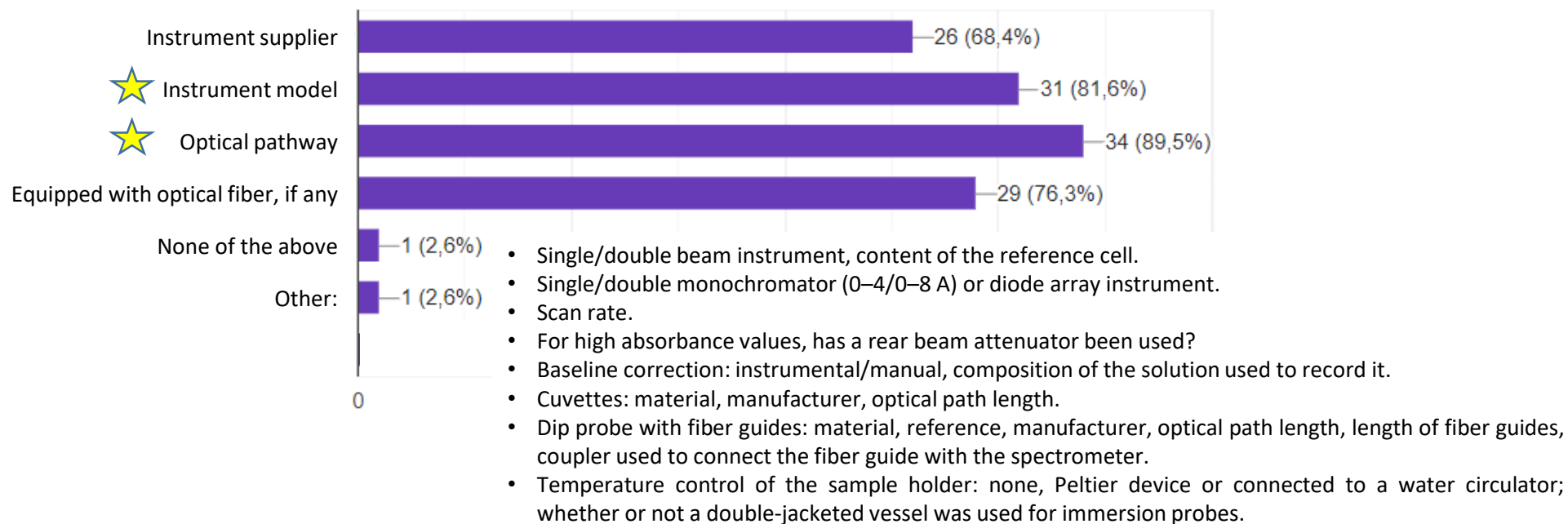
#### Other:

- Solvent, BGE, standardized acid and base solutions: all items already listed in the calibration section.
- Bare metals and metal salts: purity, trademark and batch number, recrystallization conditions, drying conditions, exact composition (co-crystallized solvent), any analytical data acquired by the users to better characterize the salt (TGA, Karl-Fischer, ICP/AAS, XRD, etc).
- Metal stock solutions: detailed preparation method (salt/bare metal dissolution), exact standardization procedure of the metal stock solutions and bibliographic references, method used to determine the exact acid concentration, storage conditions.
- Ligand: origin (purchased or synthesized), retailer and batch number, purity, purification conditions, drying conditions, exact composition any analytical data acquired by the users (the more the better, even for commercial compounds) to better characterize the ligand (CNHS/O, ion chromatography, NMR, mass data, melting point, refraction index, HPLC traces, optical rotation, UV-vis and/or IR data, TGA, Karl-Fischer, XRD etc.), weighted for each titration or aliquot taken from a stock solution, storage conditions (desiccator, temperature, under protecting gas, light protection), short/long term chemical stability (check if compounds undergoes hydrolysis/radiolysis under acidic/alkaline conditions).
- Have equilibrium conditions been reached before measurements? Potential/pH monitoring over time before starting the titration and during the experiment, other technique used to monitor the kinetics (UV-vis, etc), pH electrode drift criterion, superposition of forward and backward titrations, means used to detect precipitation (naked eye, optical monitoring, a posteriori by speciation calculations of the on-set pH value for metal hydroxide precipitation).

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### Spectrophotometry - Instrument - Spectrophotometer

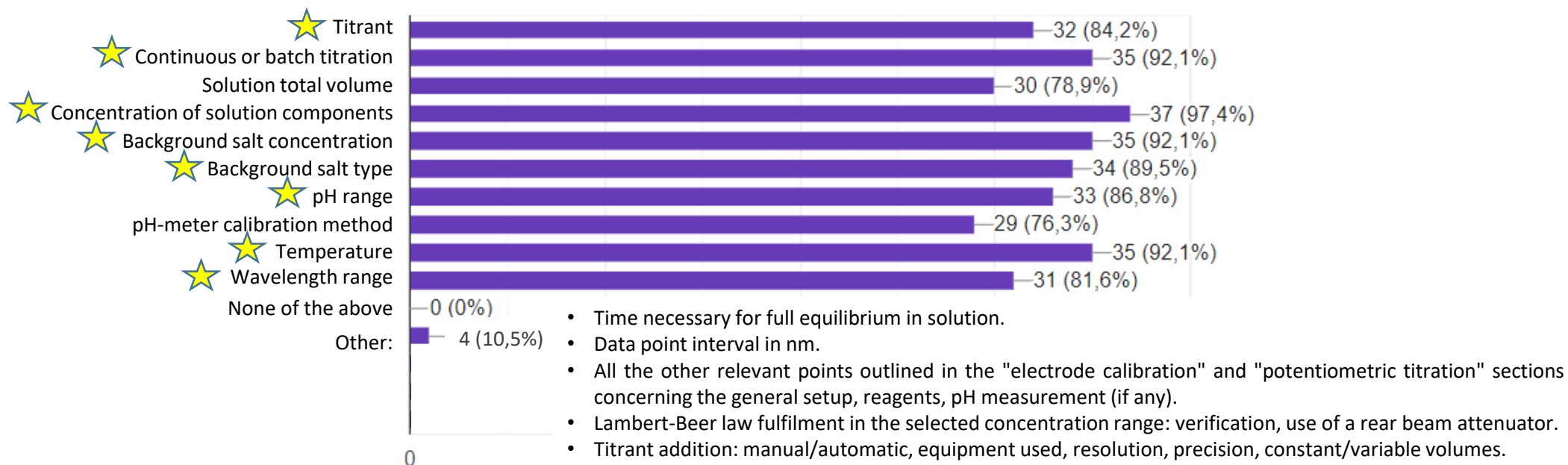
What instrumental information is necessary to describe the experiment?



## WG4 activity - NECTAR survey 2 - Answers

### Spectrophotometry - titration procedure

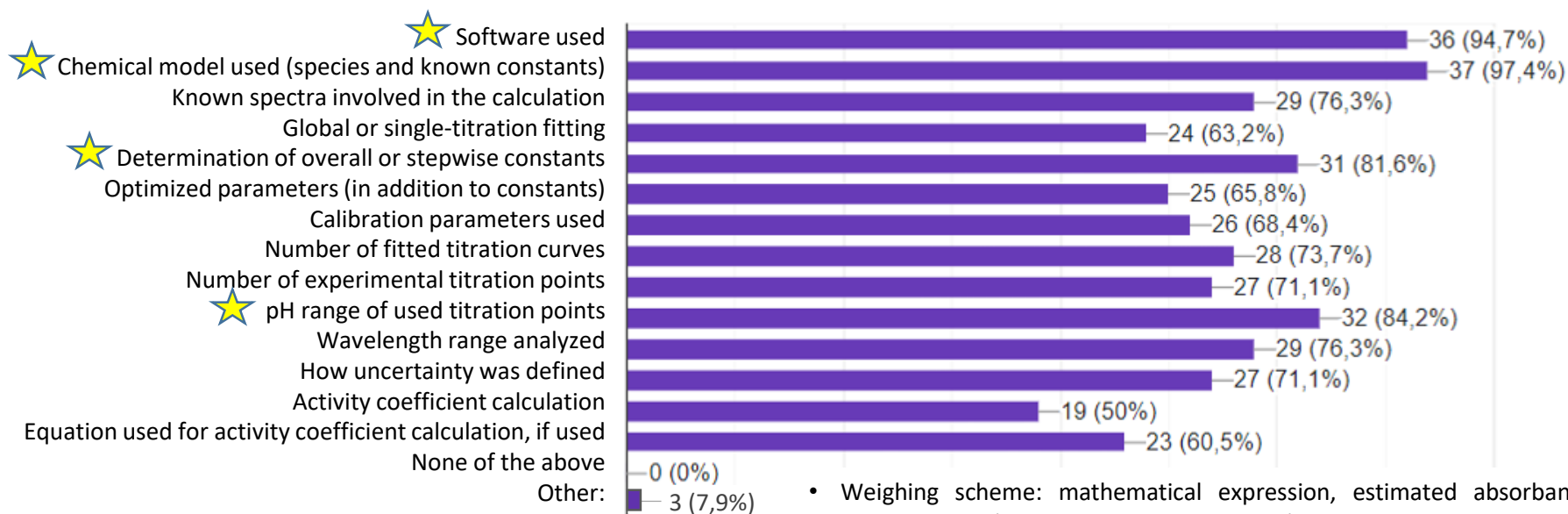
What information is necessary to describe the titration procedure used?



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### Data processing

What information is necessary to comprehensively describe the data processing?



- Weighing scheme: mathematical expression, estimated absorbance errors as a function of wavelength and concentration (values, assessment method).
- Criteria used to accept the model, goodness-of-fit parameters, plots of residuals at well-selected wavelengths and/or concentrations, plots of calculated spectra with error bars.
- Activity/concentration scale of electrode calibration.
- Assignment of calibration to titration.



Thanks for collaboration!